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New Experimental Method for *In Situ* Determination of Material Textures at
Simultaneous High-Pressure and –Temperature by Means of Radial
Diffraction in the Diamond Anvil Cell.

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Abstract

We introduce the design and capabilities of a new resistive heated diamond anvil cell that can be used for side diffraction at simultaneous high-pressure and -temperature. The device can be used to study lattice-preferred orientations in polycrystalline samples up to temperatures of 1100 K and pressures of 36 GPa. Capabilities of the instrument are demonstrated with preliminary results on the development of textures in the BCC, FCC and HCP polymorphs of iron during a non-hydrostatic compression experiment at simultaneous high-pressure and -temperature.

I. Introduction

For many years, Diamond Anvil Cell's (DAC) have been used to determine the compression behavior of materials at high-pressure¹. Of particular interest has been the use of the DAC to determine the stress and strain state of minerals and compounds under non hydrostatic conditions and ambient temperatures using the radial diffraction geometry²⁻⁶ (Fig. 1). Most of these experiments are only possible because of the use of x-ray transparent gaskets, consisting of amorphous B inserts⁶⁻⁹ or Be³⁻⁴ that allow the collection of x-ray diffraction data from samples perpendicular to the loading axis. Radial diffraction has also been used to study texture and lattice preferred orientations (LPO) in plastically deformed materials¹⁰ by measuring intensity variations along the Debye rings. For instance, texture information was recently used to model the activation of slip planes at high-pressure in geological relevant materials such as polycrystalline $(\text{Mg}_{0.9}\text{Fe}_{0.1})\text{SiO}_3$ post-perovskite¹¹ and its Ge analogue¹². However, extrapolation of these finding to high-pressure and -temperature conditions of the Earth lower mantle have proven to be incomplete because of our lack in understanding the effect of temperature on high pressure plasticity.

There are multiple avenues one might take to study plastic deformation under simultaneous high-pressure, -temperature while probing the sample using *in-situ* x-ray diffraction: a) in a Large Volume Press (LVP) such a Drickamer¹³ or the multi anvil device¹⁴ b) in a DAC that is heated internally with a laser¹⁵, and c) in a DAC that is heated externally through a resistive heater such as graphite. Each of these techniques has its advantages and disadvantages. While there has been significant progress using

different types of LVP to measure stress and textures at simultaneous high-pressures and –temperatures, it is currently not possible to use those instruments to perform quantitative deformation experiments beyond pressures of the transition zone (21 GPa and 2000 K). Recently, radial diffraction experiments in the DAC using in situ laser heating have made significant progress¹⁵⁻¹⁶. They are, however, complicated by the presence of large temperature gradients that develop during heating. To this day, externally heated DAC have not been used for stress and texture analyses because the resistive heater would lie in the x-ray path. A design of a cell capable of such experiments was published recently¹⁷ but has not been tested for radial diffraction experiments. Furthermore, gasket materials typically used in radial diffraction experiments lose their strength at high temperatures (as do normal diamond anvils) and thus significantly limit the pressure and temperature range accessible. In addition, accurate measurement of pressure at high-temperature has been an ongoing problem in resistive heated DAC but is now actively being addressed by the community¹⁸⁻²². In contrast to the other two techniques motioned above, the externally heated DAC experiment offers good temperature and pressure control and can reach much higher pressures than the LVP. Furthermore, the development of sintered nano-diamond anvils could significantly expand the accessible pressure temperature range because the high temperature strength of the anvils can be significantly improved²³⁻²⁴. In the future, a combination of experiments on large and well-controlled samples in a resistively heated radial diffraction setup and higher temperature laser-heated measurements will allow the study of plasticity at pressures and temperatures relevant to the Earth deeper mantle. More generally, the technique could also be useful to study the

plasticity of materials relevant to various branches of material sciences, such as the study of super-hard materials for which *in-situ* characterization of strength and plastic properties are absolutely necessary²⁵⁻²⁷.

In this paper, we present a new radial diffraction technique that allows measurement of textures and stress at simultaneous high-pressure and –temperatures in the DAC using an external resistive graphite heater. We demonstrate the current capability of the technique by measuring textures of Fe and its high pressure polymorphs at simultaneous high-pressure and –temperatures.

II. Experimental Method

Resistive-heated DAC for Radial Diffraction X-ray (RDX) experiments

The new technique presented here allows us to perform RDX at simultaneous high-pressures and –temperatures. It combines three existing techniques, a) amorphous boron gaskets that allows radial access to the sample⁹, b) externally graphite heated gaskets²⁸ and c) membrane devices for accurate control of pressure²⁹.

In order to accommodate the resistive heating setup and membrane control we designed a new Mao-Bell type DAC (Fig. 2 A and B) that will be called Resistive-Heated-Radial-X-ray-Diffraction-Diamond-Anvil-Cell (RH-RDX-DAC). In contrast to a conventional Mao-Bell type cell, the new cell has openings on two sides that allow collection of diffraction patterns perpendicular to the loading axis.

Heating of the sample is achieved by using graphite heaters that surround the gasket assembly. The front of the cell has been further modified to accept two molybdenum rods that supply power to the graphite heater. The ends of the molybdenum rods are machined to a perpendicular extension with a step to hold the graphite heater in place, when the piston of the DAC presses against the graphite. The graphite heater consists of two pieces of flexible graphite foil with 1mm thickness (Alfa Aesar, 97% pure, metal basis) that sandwiches the gasket assembly. The graphite and the gasket assembly are isolated from the tungsten carbide seats by a thin disk of sintered alumina (McDanel Advanced Ceramics Technologies LLC, 99% purity). Prior to sample loading the graphite foil is indented between the diamonds with a thick stainless steel gasket in place of the final gasket assembly. This process shapes the graphite foil, provides a tight

contact between the diamonds and the graphite, and allows enough space to position the final gasket assembly.

The gasket consists of a 50 μm thick amorphous boron gasket insert⁹, 400 μm in diameter, with a 50 μm diameter hole that serves as sample chamber (Fig. 3). The kapton sheet that has been used to support the boron insert in RDX experiments at 300 K is replaced by a fluorine bearing mica sheet of 100 μm thickness (H. C. Materials-Corporation). The melting temperatures of this fluorine mica (1373 K) is significantly higher than the maximum working temperature of kapton (~ 500 K), extending the accessible temperature range in the experiment. Moreover, this material forms a single crystal with a strongly anisotropic crystal structure whose contribution to the diffraction is being limited because the sheet structure is positioned parallel to the incoming beam. Finally, a small channel is cut into the graphite heater along the path of the incident beam to avoid significant diffraction from graphite.

In order to prevent the piston and the cylinder to stick to one another at high-temperature and thus preventing the in- and decrease of the pressure during the experiment, we use Molykote 1000 Paste (Dow Corning) as a lubricant and heated the outside of the cylinder of the DAC with a strip heater to a maximum of 673 K.

Fine pressure tuning in the cell is provided by adapting the RH-RDX-DAC with a membrane similar to that developed by the high-pressure group at Lawrence Livermore National Laboratory (LLNL) allowing a control of the pressure remotely during the experiment.

In-situ x-ray diffraction

We performed in-situ x-ray diffraction at beamline 16BMD of the High-Pressure Collaboration Access Team (HPCAT), Advanced Photon Source (APS), Argonne National Laboratory (ANL). In the experiment the wavelength of the 15x15 μm monochromatic x-ray beam was tuned to 0.38745 Å at which it offered a optimal flux of $\sim 2 \times 10^8$ photons/sec and resulted in a collection time of 15 min. Data were collected on a 3450 x 3450 pixel Mar 345 image plate. Sample to detector distance (285.943 mm), detector tilt, and pixel size ratios were calibrated using a CeO₂ standard from NIST (674b).

Sample was a pure commercial powder of iron (Alfa Aesar, 99.9 % purity) with a starting grain size of 1 to 2 μm . In this run, a small piece of 5 μm thick gold foil was added for additional pressure calibration but could not be seen on the x-ray diffraction images.

Temperature in the radial diffraction DAC

The graphite foil was heated through an ultra stable DC power supply from Hewlett Packard with a power range of 0 to 8 V and 0 to 220 A (Type A6607). Power was increased remotely through an external voltage control provided by EPICS (Experimenting Physics Industrial Control System) at the beamline control area.

Temperature was measured with two Pt-Pt30%Rh (Type R) thermocouples. The first thermocouple was positioned on the diamond facet of the anvil on the cylinder side of the cell, about half way to the tip. The second thermocouple was located between the

two pieces of graphite foil, close to the fluorine bearing mica (Fig. 1). The thermocouple voltages (EMF) were measured with a Keithley 7200 equipped with an internal cold junction correction board. The accuracy of the thermocouple reading is +/-5 K.

Data processing

Diffraction images were quantitatively analyzed for cell parameters, phase proportions, texture, and lattice strains using the Rietveld method as implemented in the software package MAUD³⁰ according to the procedure described in³¹. Diffraction images were integrated over 5° increments of the azimuth angle resulting in 72 slices. Data were fit using a BCC, FCC and HCP structure with adjustable phase ratios. The variations of peak positions with azimuth were adjusted assuming

$$d_m(hkl, \psi) = d_p(hkl) [1 + (1 - \cos^2 \psi) Q(hkl)]$$

where ψ represents the angle between the diffracting plane normal and the maximum stress direction, $d_m(hkl, \psi)$ the measured d-spacing for the (hkl) line diffraction at ψ , $d_p(hkl)$ is the hydrostatic d-spacing of the (hkl) line and is calculated from the hydrostatic lattice parameters, and $Q(hkl)$ is the lattice strain parameters. Values of $Q(hkl)$ can then be used to analyze the stress state of the sample using elastic lattice strain theories³² or elasto-pastic self consistent models³³. LPO were fitted using the E-WIMW algorithm assuming cylindrical symmetry about the compression direction and using an orientations distributions functions (ODF) resolution of 7.5°.

Refinements for the BCC phase were based on measurements on the 110, 200, and 211 diffraction lines. Refinements for the FCC phase were based on measurements

on the 111, 200, and 220 diffraction lines. Refinements for the HCP phase were based on measurements on the $10\bar{1}0$, 0002, $10\bar{1}1$, $10\bar{1}2$, and $11\bar{2}0$ diffraction lines.

Pressure at high-temperatures

Measuring pressure at high-temperatures has been a long standing problem for resistive heated experiments. Spectroscopic methods have been used to determine the accurate pressure using fluorescence shifts in Ruby, $\text{SrB}_4\text{O}_7\text{:Sm}^{2+}$ or YAG crystals¹⁸⁻²⁰. More recently shifts of the first order Raman peaks of diamond³⁴ and cubic boron nitride²⁰ at high-temperature have also been used. Alternatively, one may determine pressure using a P-V-T thermal Equation of State (EoS) of an internal standard such as Au or Pt. The later has been established by using multiple pressure calibrants to produce an internally consistent set of high-pressure and temperature EoS²¹⁻²².

In the present experiment, we decided to calibrate pressures using x-ray diffraction of an internal standard. Since gold could not be seen in our diffraction patterns (even at 300 K and 1 bar), we decided to use the equation of state of the sample itself, iron, in the BCC structure³⁵, and later HCP structure³⁶, to estimate pressure at any given temperature during the experiment. To correct for the effect of non-hydrostatic stresses, pressures were calculated using the hydrostatic cell parameters of BCC and HCP iron as derived from the MAUD refinements. Fortunately, in the entire pressures and temperatures range covered in this run, either BCC or HCP did coexist beyond their stability limit and allowed us to calibrate pressure within the field of stability of the FCC

phase. The temperature pressure path explored during the experiment can be seen in Fig.

5.

III. Results and discussion

Accuracy of temperature measurements in the RH-RDX-DAC

Because of the high thermal conductivity of diamonds and complexity of the sample assembly, temperature measurements with thermocouples that do not directly surround the sample have been questioned in the past³⁷. To investigate this issue, we measured the melting temperature of Pb (605K), Al (933.2 K) and NaCl (1077 K) in a second membrane controlled graphite resistively-heated DAC that was used recently²⁸. This membrane cell uses the exact same heating assembly and similar thermocouple positions than those described in this work. Thus, it represents an accurate test of our capability to measure temperatures in the low to medium temperature range.

Melting was observed optically through a Navitar zoom microscope with a maximum primary magnification of 20x. Samples of Al, and Pb were separated in the sample chamber by NaCl to avoid reaction between the different standards during heating. Sample chamber was illuminated by reflected and transmitted light. Onset of melting was determined by observing the appearance of movements in the liquid phase and changes in the sample surface texture. Both approaches are commonly used to determine melting of metals in laser heated DAC experiments with accuracy in the ten's K³⁸.

Temperatures recorded on both thermocouples at zero pressure are plotted as a function of power in Fig. 4 A. Up to 1077 K the discrepancy between the temperature recorded at the onset of melting and the melting temperature reported in the literature are

within +2 to -30 K for the thermocouple positioned between the two pieces of graphite foil and up to 60 K for the thermocouple positioned on the facet of the diamond.

These measurements imply that temperatures measured by the thermocouple positioned between the two graphite foils are within 30 K of the real temperature. Note that the difference between melting temperatures reported in the literature and those measured by the second thermocouple, positioned about 1 mm away from the diamond tip, only extend to +60 K. This relatively small difference indicates that the gasket assembly and top part of the diamond is very efficiently heated by the graphite foil and that temperature gradients in the gasket, sample, and diamond tip area are relatively small.

Temperatures measured in both thermocouples during the RH-RDX-DAC experiment on iron are plotted as a function of power in Fig. 4 B). In contrast to the melting experiment above, temperatures recorded by the thermocouple positioned between the two graphite foils are always higher than those recorded at the facet of the diamond. This difference can be explained by the fact that the thermocouple at the facet of the diamond is not always positioned precisely the same way. In addition we find that the shape of the WC seat, i.e. the opening of the seat, significantly affects the amount of heat being carried away from the back of the diamond anvil. The opening of the seat in the DAC used for the melting experiment is much smaller than that of the radial diffraction RH-RDX-DAC seats. Hence, the temperatures recorded at the facet of the diamond anvil of the radial diffraction cell are lower. Nevertheless, the difference in

temperatures recorded by the two thermocouples never exceeds 62 K. This indicates a fairly homogeneous heating during the experiment.

Phase diagram and equation of state of FCC-iron

Table 1 lists the refined cell parameters obtained from positions of the diffraction peaks of BCC-, FCC-, and HCP-iron as a function of pressure and temperature during the experiment. We obtain small errors for the cell parameters of BCC-iron before the appearance of other iron phases but rather large errors later on. This is due to inconsistencies between the cell parameters obtained from various diffraction lines, which could be due to the presence of a mixed phase.

In our experiment, the transition from the BCC to the FCC phase of iron occurred at temperatures higher than those reported in the literature^{36, 39-40} (Fig. 5). However, phase transitions in iron are known to be very sensitive to both kinetics³⁶ and stress⁴¹. Our experiment was designed to prove the capabilities of the instrument. As such, we did not perform multiple transition cycles and long heating periods necessary for full phase conversions and proper phase diagram analysis. Therefore, small discrepancies between our experiments and reported phase boundaries can be attributed to kinetics and stress.

Our cell parameters for FCC-Fe are also significantly different from those reported in the literature⁴⁰⁻⁴¹. This is particularly true at low pressures. Differences between our measured cell parameters and the published EOS⁴⁰ decrease with increasing pressure. It is known that coexistence of two or three phases associated with a phase transition can cause volume anomalies due to the difference in linear compressibility and

thermal expansivity. This can probably explain the observed discrepancy. Proper measurement of the EOS of FCC-Fe should be performed with better phase conversion, longer heating periods, and minimal stress conditions.

Diffraction and texture development in iron at high-pressure and temperature

The greatest challenge collecting and processing radial diffraction data from a DAC originates from the background created by material that lies in the diffraction path. In the case of the HT-RDX-DAC, graphite, fluorine mica and amorphous boron contribute to the background (Fig. 1).

Fig. 6 shows the diffraction image collected at 976 K and 12.5 GPa. The diffraction image clearly shows powder diffraction peaks corresponding to the 111, 200 and 220 lines of FCC-iron and 110, 200, and 211 lines of BCC-Fe. Diffraction from graphite is limited to the most intense 002 peak. Single crystal spots of mica are significant but localized; they can be easily filtered out during the processing. Due to grain growth and recrystallization, iron diffraction lines are spotty. Nevertheless, variations of diffraction intensities with orientation can be observed. They are related to LPO in the polycrystalline sample. Similarly, slight variations of peak positions with orientations can be used to model the differential stress supported by the sample.

Reliable textures can be extracted. Fig. 7, for instance, presents inverse pole figure of the compression direction obtained for BCC- and FCC-iron at 12.5 GPa and 976 K and FCC- and HCP-iron at 33.2 GPa and 899K. Orientations distributions functions

(ODF) were obtained directly in MAUD and further smoothed with a 10° gauss filter in Beartex⁴².

Upon compression, BCC- iron develops a mixed {100} and {111} texture that is compatible with previous observations on BCC-iron⁴³ and interpreted as slip along {110}<111>. When BCC- iron is heated between 295 and 976 K, we observe a recrystallization of the sample, as evident by the spotty pattern in Fig. 6. The {100} and {111} maxima are preserved during heating.

Texture obtained after formation of FCC-iron show a maximum near {110} with minima near {100} and {111}. This texture is typical for FCC metals in compression with slip on {111}<110>, but could also be related to texture being transmitted from the BCC to the FCC phase according to the Kurdjumov-Sachs orientation relations⁴⁴. Further study and processing will be needed to separate the two effects.

Textures obtained in HCP-Fe seem different from those reported before^{5, 16, 34}. Further study and modeling will be required for full interpretation, which goes beyond the scope of this paper.

Application of the instrumentation

One of the primary applications of radial diffraction DAC experiments is the investigation of deformation mechanisms and possible preferred orientation patterns of rocks in the deep Earth, e.g. the lower mantle and the core. Today, radial diffraction is the only direct method to probe plastic processes at pressures of the Earth lower mantle. Until now, an obvious limitation in using ambient temperature results of LPO formed in the DAC was the lack of temperature. It was therefore desirable to develop reliable heating methods for radial diffraction. The technique presented here will allow the investigation of plastic properties of minerals up to temperatures of about 1500K, a temperature at which diffusion processes that are critical for plasticity can become active.

The instrumentation presented here can also be used for the study of strength of newly designed super-hard materials. Most of these studies are limited to measurements of quasi-hydrostatic equations of state for the extraction isothermal bulk moduli. This is insufficient to assess the strength and behavior of the material under complex conditions. Using the above instrumentation, one could study the strength and microscopic plastic deformation mechanisms under various conditions of pressure and temperature. This will be valuable information for understanding the plastic behavior of industrially relevant materials.

VI. Conclusion

We designed a new DAC experimental setup that allows the collection of x-ray diffraction data in a radial geometry from samples plastically deformed at simultaneous high pressure and high temperature. The setup was successfully tested up to temperatures of 1100 K and pressures of 36 GPa.

Errors in temperature measurements are within ± 30 K in the entire temperature range explored. Moreover, temperatures measured ~ 1 mm away from the tip of the diamond, on the diamond facet show a temperature difference of only ± 60 K, indicating that the gasket and tips of the diamonds are heated very homogeneously.

The instrumentation allows the collection of radial x-ray diffraction data of sufficient quality for the extraction of texture and stress information using the Rietveld refinement program “MAUD”.

Detailed analysis of the data presented here will be discussed elsewhere. However, preliminary analysis indicates the presence of a strong $\{110\}$ texture in FCC-iron, compressed in the diamond anvil cell between 7.1 and 12.5 GPa at 970K, after the transformation from BCC-iron.

This new instrumentation can now be used for the study of plastic properties of minerals and other materials at simultaneous high-pressure and –temperatures.

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Table:

Table 1: Cell parameters refined from peak positions of BCC, FCC and HCP.

Temperature was measured with thermocouple. Pressure was calculated using the cell parameters of BCC and HCP iron and their respective high-pressure and –temperature EoS (Komabayashi et al 2009 EOS, Uchida et al 2001). The error in the temperature measurement is estimated to be +/- 30 K and the error in the pressure calculation is +/- 0.7 GPa based on an average error of 0.004 Å in the cell parameters. Errors for the cell parameters obtained for BCC-Fe before the appearance of other phases are approximately 0.001 Å. When multi-phase aggregate is present, errors on cell parameters are on the order of 0.004 Å.

| Image | T (K) | P (GPa) | Fe (BCC) a (Å) | Fe (FCC) a (Å) | Fe (HCP) a (Å) c (Å) | |
|-------|----------|------------|-------------------|-------------------|-------------------------|-------|
| 3 | 295 | 0,1 | 2,866 | | | |
| 13 | 295 | 0,2 | 2,865 | | | |
| 22 | 295 | 1,5 | 2,858 | | | |
| 24 | 295 | 3,4 | 2,848 | | | |
| 28 | 295 | 4,6 | 2,842 | | | |
| 31 | 295 | 4,8 | 2,841 | | | |
| 35 | 373 | 12,8 | 2,807 | | 2,438 | 4,028 |
| 36 | 374 | 9,1 | 2,823 | | 2,454 | 3,978 |
| 37 | 374 | 5,9 | 2,838 | | 2,468 | 3,992 |
| 38 | 373 | 4,8 | 2,843 | | 2,473 | 4,096 |
| 40 | 474 | 8,6 | 2,828 | | 2,464 | 4,118 |
| 41 | 473 | 8,2 | 2,830 | | 2,468 | 4,117 |
| 42 | 474 | 6,9 | 2,836 | | 2,473 | 4,136 |
| 43 | 474 | 5,1 | 2,845 | | 2,477 | 4,151 |
| 45 | 570 | 4,1 | 2,853 | | 2,497 | 4,180 |
| 46 | 672 | 1,9 | 2,869 | | | |
| 47 | 673 | 3,8 | 2,858 | | | |
| 48 | 774 | 1,0 | 2,878 | | | |
| 49 | 773 | 3,6 | 2,863 | | | |
| 50 | 773 | 5,6 | 2,852 | | | |
| 51 | 773 | 7,0 | 2,845 | | | |
| 53 | 878 | 5,5 | 2,856 | | | |
| 54 | 873 | 7,0 | 2,848 | | | |
| 56 | 973 | 7,1 | 2,851 | 3,619 | | |
| 57 | 973 | 8,3 | 2,845 | 3,606 | | |
| 58 | 974 | 10,3 | 2,835 | 3,580 | | |
| 59 | 973 | 11,2 | 2,831 | 3,566 | | |
| 60 | 972 | 11,6 | 2,829 | 3,558 | | |
| 62 | 976 | 12,5 | 2,825 | 3,546 | | |
| 63 | 974 | 14,5 | 2,818 | 3,529 | 2,481 | 4,030 |
| 64 | 973 | 16,4 | | 3,519 | 2,476 | 4,012 |
| 65 | 975 | 18,0 | | 3,508 | 2,472 | 3,993 |
| 66 | 976 | 18,5 | | 3,504 | 2,470 | 3,990 |
| 67 | 976 | 19,9 | | 3,498 | 2,465 | 3,981 |
| 68 | 976 | 21,3 | | 3,491 | 2,460 | 3,970 |
| 69 | 976 | 22,9 | | 3,484 | 2,455 | 3,959 |
| 70 | 980 | 24,0 | | 3,476 | 2,451 | 3,953 |
| 71 | 953 | 25,2 | | 3,472 | 2,446 | 3,944 |
| 72 | 958 | 25,0 | | 3,470 | 2,447 | 3,946 |
| 73 | 958 | 25,6 | | 3,470 | 2,445 | 3,942 |
| 74 | 959 | 26,6 | | 3,465 | 2,442 | 3,936 |
| 75 | 938 | 26,4 | | 3,465 | 2,442 | 3,936 |
| 76 | 923 | 27,8 | | 3,465 | 2,438 | 3,924 |
| 77 | 903 | 28,2 | | 3,459 | 2,436 | 3,922 |
| 78 | 902 | 28,4 | | 3,455 | 2,435 | 3,921 |
| 79 | 901 | 29,4 | | 3,453 | 2,432 | 3,916 |
| 80 | 900 | 31,0 | | 3,444 | 2,427 | 3,908 |
| 81 | 899 | 33,2 | | 3,434 | 2,421 | 3,897 |
| 83 | 821 | 36,6 | | 3,419 | 2,410 | 3,876 |
| 85 | 707 | 35,4 | | 3,419 | 2,410 | 3,877 |
| 87 | 555 | 36,1 | | 3,413 | 2,406 | 3,865 |
| 88 | 422 | 36,0 | | 3,409 | 2,404 | 3,860 |
| 89 | 313 | 36,2 | | 3,410 | 2,402 | 3,855 |

Figure Caption:

Figure 1: Setup of the RH-RDX-DAC experiment. The polycrystalline sample is confined under non-hydrostatic stress between the two diamond anvils. A monochromatic x-ray beam is sent through the gasket, perpendicular to the compression direction. Microscopic properties of the sample are studied with x-ray diffraction by analyzing the variation of the diffraction spectra as a function of the azimuthal angle δ .

Figure 2: 3D cross section (A) and image (B) of the new RH-RDX-DAC for radial diffraction at simultaneous high-pressure and –temperature.

Figure 3: Gasket assembly consisting of an amorphous boron disk positioned on the diamonds anvils and surrounded by a fluorine mica for support of the insert.

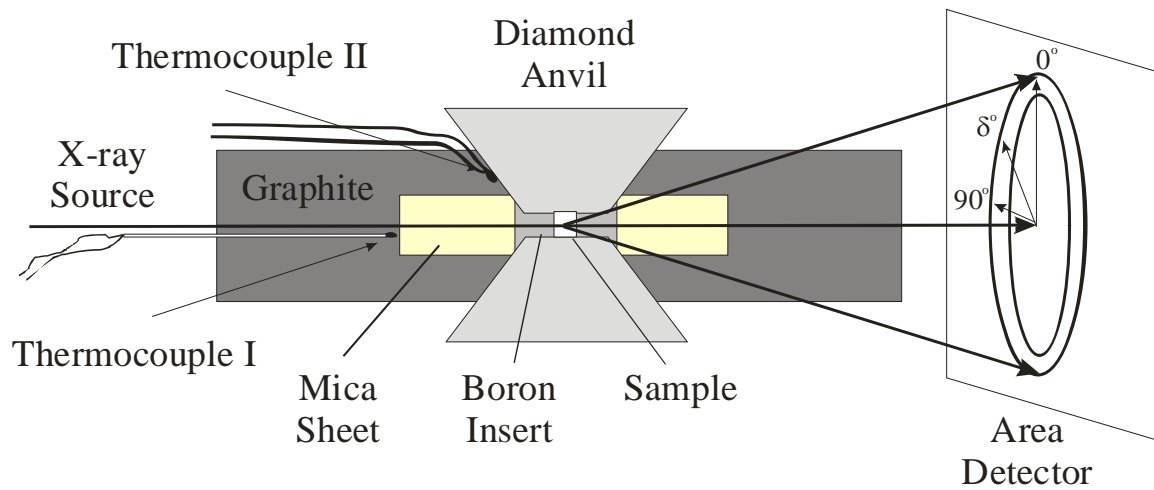
Figure 4: A) Ambient pressure temperature measurements as a function of power in the RH-DAC. Onset of melting was observed optically through a microscope and are indicated by arrows on the figure. Differences between the melting temperatures reported in the literature and those measured using the thermocouple positioned between the graphite foil for Pb, Al and NaCl are -22, 2 and 30 K, respectively.

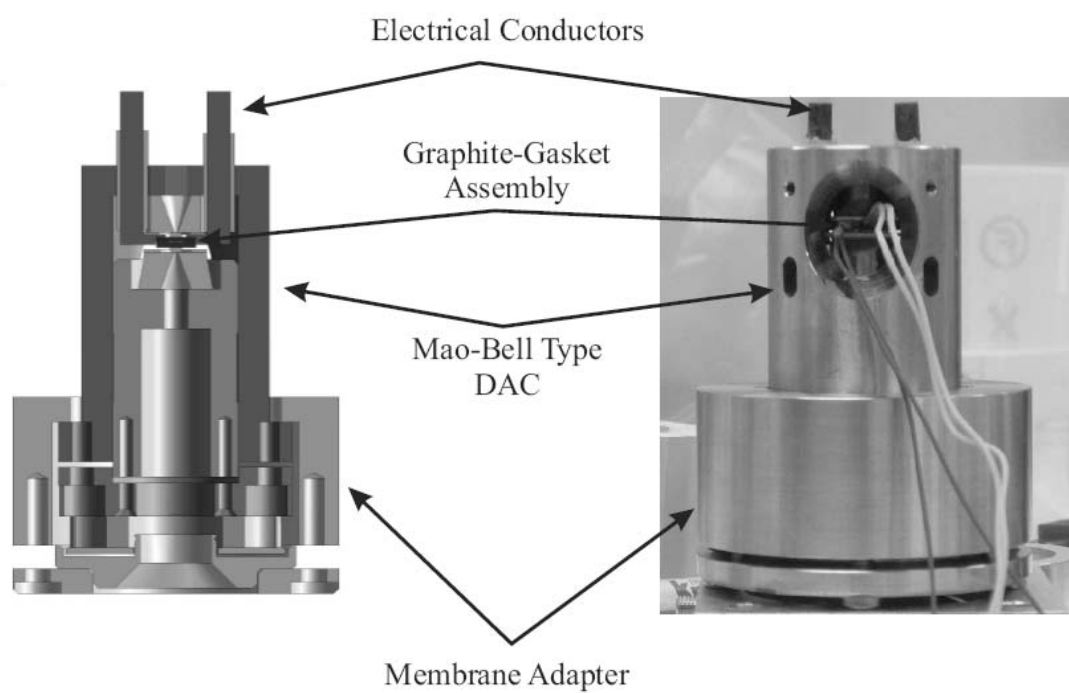
Figure 4: B) High pressure temperature measurements as a function of power in the radial diffraction membrane driven Mao-Bell type DAC. The maximum difference between the temperatures recorded by both thermocouples (see text) is 66 K recorded at the highest temperature.

Figure 5: Pressure-Temperature path explored during the second radial diffraction experiment. Grey circles denote the presence of α -Fe, open squares ϵ -Fe, and black circles γ -Fe. γ -Fe coexists with ϵ -Fe past its stability limit until the end of the experiment. Phases in the sample were identified from the diffraction images. Solid and dotted lines are the phase Fe diagram upon compression and decompression, respectively, as published in (Uchida et al 2001). Dash-dotted line the ϵ - γ phase boundary of (Komabayashi et al 2009 PD).

Figure 6: Radial diffraction image A) and unrolled diffraction image B) of FCC iron at 12.5 GPa and 976 K and in the graphite resistive heated Mao Bell type DAC. Besides diffraction peaks of FCC (111, 200 and 220) and BCC-Fe (110, 200, 211), one can see powder diffraction peaks of graphite (002) and single crystal diffraction spots of fluorine bearing mica.

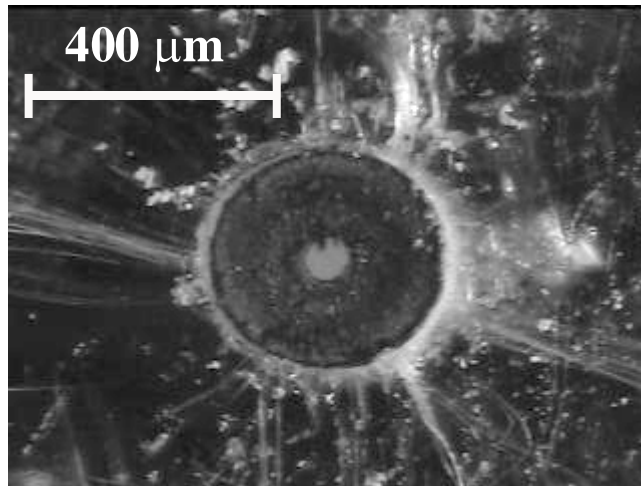
Figure 7: Inverse pole figure of the compression direction for BCC- and FCC-iron at 12.5 GPa and 976 K (A,B) and FCC- and HCP-iron at 33.2 GPa and 899K (C,D). Equal area projection. Linear scale in m.r.d.

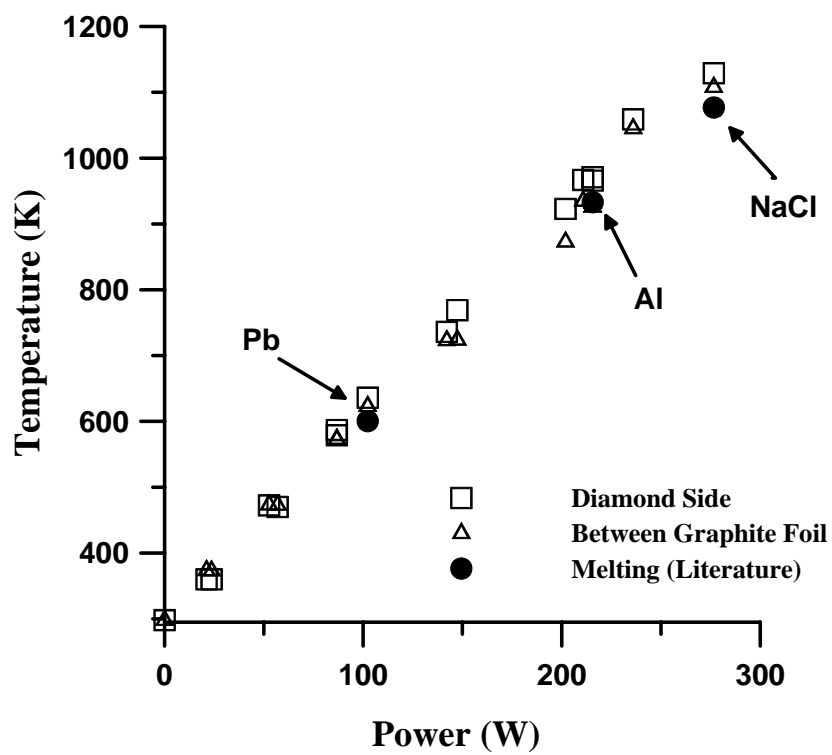




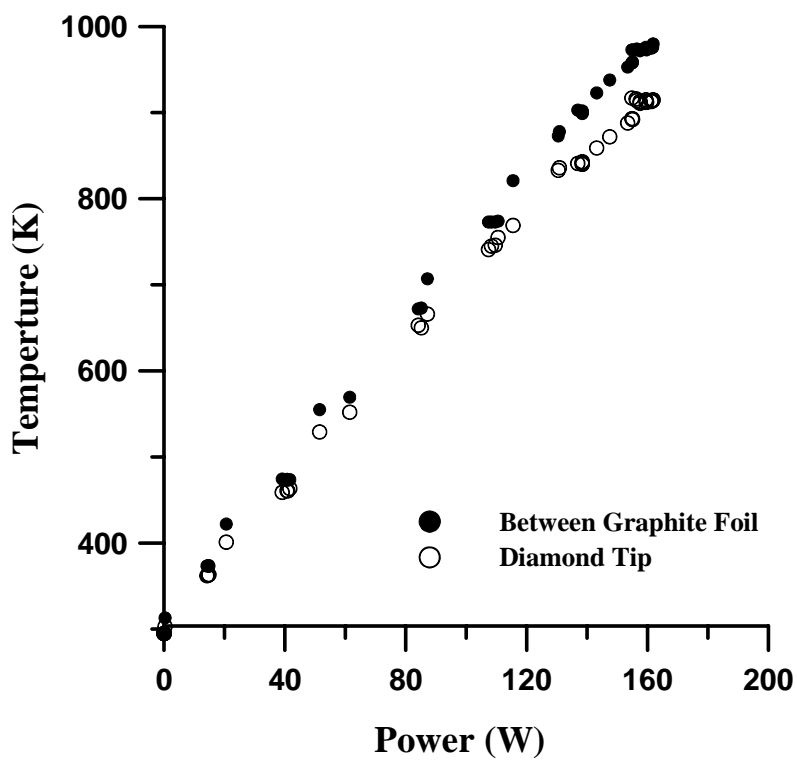
A

B





A)



B)

